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Docket No. 968-20-003

2814

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Zinc Matrix Power, Inc.

Serial No. 09/839,324

Art Unit: 1745

Filed: April 19, 2001

Examiner: Jonathan S. Crepeau

Title: ALKALI RESISTANT CELLULOSE SEPARATOR

TERMINAL DISCLAIMER

Mail Stop Non-Fee Amendment

Commissioner for Patents

P.O. Box 1450

Alexandria, VA 22313-1450

Sir:

I, Skip Zeiler, the President of Zinc Matrix Power, Inc. (hereinafter Zinc Matrix Power), 121 East Mason Street, Santa Barbara, California, represent that Zinc Matrix Power is the assignee and the exclusive owner of the entire right, title, and interest of, in and to application no. 09/839,276 filed on April 19, 2001 for RECOMBINANT SEPARATOR, as indicated by the records of the United States Patent and Trademark Office at 011733/0643.

The undersigned has reviewed all the documents in the chain of title of the patent application identified above and, to the best of undersigned's knowledge and belief, title is in the assignee identified above.

Zinc Matrix Power hereby disclaims the terminal part of any patent granted on the above-identified application, which would extend beyond the expiration date of any U.S. patent to be granted on copending application 09/839,324 filed April 19, 2001 also assigned to and owned by Zinc Matrix Power as indicated by the Assignment recorded at 011729/0623 and hereby agrees that any patent so granted on the above-identified application shall be enforceable only for and during such period that the legal title to said patent shall be the same as the legal title to any United States Patent to be granted on the copending application, this agreement to run with any patent granted on the above-identified application and to be binding upon the grantee, its successor or assigns.

ZINC MATRIX POWER, INC.

By:

Skip Zeiler, President

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of Zinc Matrix Power, Inc.

Serial No: 09/839,324

Law Office: 1746

Filed: April 19, 2001

Examiner: Jonathan Crepeau

Mail Stop Fee  
P.O. Box 1450  
Commissioner for Patents  
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 CFR 1.132

Wilson Hago, declares and states that:

1. He was awarded an A.B. in Physics from Harvard University in 1985, and a Ph.D. in Chemistry from Brown University in 1994.

2. He is the author of 3 articles, and the applicant of 4 patent applications and the patentee of 3 patents as listed in the Curriculum Vitae attached to this Declaration.

3. He has spent 6 years in the research and development of cellulosic compositions, reactions and products and is considered an expert in cellulosic chemistry.

4. The nucleophilic reaction described in the

Specification necessarily results in saturated hydrocarbon linkages such as alkylene from the reaction of dihaloalkanes with cellulose hydroxyl groups and inorganic base.

5. He is an inventor of the above-identified application.

6. He has studied the Office Action dated February 10, 2003 and the cited references and does not believe the references disclose nor render the present invention obvious.

7. The present invention discloses a battery separator that improves on the native strength of regenerated cellulose and is more resistant to oxidation than regenerated cellulose. The invention proceeds by crosslinking dissolved cellulose and forming a film from this crosslinked cellulose.

Cellulose, with a degree of polymerization from 200 to 1200, in the form of, but not limited to, microcrystalline cellulose, cotton fiber, paper and microgranular cellulose, is dissolved using a variety of different solvents, including, but not limited to, LiCl/DMAC, trifluoroacetic acid and N-morpholine N-oxide. With LiCl/DMAC, the preferred range is 3 to 8% wt LiCl to DMAC and the applicable range for the percent weight solution of cellulose to solvent is 1 to 11%.

After dissolving the cellulose, the hydroxyl groups on the cellulose are deprotonated by adding an amount of a base, in particular an inorganic hydroxide such as NaOH. The base is added in sufficient quantity to deprotonate just a small fraction of available hydroxyls, usually 1 to 10%, for an excess would result in side reactions causing chain cleavage with consequent depolymerization and degradation in film strength.

After the deprotonation is complete, a dihalide containing 4 to 6 carbon atoms is introduced into the reaction vessel. The dihalide reacts with deprotonated sites on adjacent cellulose chains to form hydrocarbon cross-link groups. The crosslink reaction preferable conducted at temperatures from 55

to 90 degrees Celsius for periods ranging from 8 to 24 hours. The amount of dihalide added is the gram-equivalent to all of the hydroxyls available. The NaOH is allowed to settle and the solution is then cast via conventional methods. These methods are known to those skilled in the art of membrane fabrication.

They include extrusion of the solution onto a conveyor belt, casting onto a glass plate with a casting knife or casting onto a well-leveled glass plate to form a separator having a thickness from 10 microns to 250 microns.

After casting, the resulting solution is coagulated with conventional techniques, preferably using water as the coagulating agent. Coagulation may be attained either by exposure to ambient moisture or by direct application of a water stream to the resulting solution. The coagulated cellulose material is washed to remove the solvent and the salt. It is possible to employ alcohols mixed with water, but it is preferable that they be kept below 50% by volume.

After thorough washing of the resulting gel, the gel may be dried with any conventional technique such as air drying, vacuum drying or press drying.

The saturated hydrocarbon cross-linked cellulose is stronger than cross linked cellulosic linkages formed by addition reactions of saturated olefin cross linking agents because olefin-based methods tend to produce weak separators because radical hydroxyl groups competitively attach sugar rings. Further, the cellulosic linkages formed by ionic reaction have better ionic conductivity and resistance to oxidation.

8. SU651436 only discloses a silver-zinc battery containing cross-linked cellulose. Klug teaches a gelled fuel composition. The references are from unrelated fields and there is no need or suggestion in the Swiss reference to lead one skilled in the art to Klug. Furthermore, the cross-linking agent is an olefin, i.e. vinyl benzyl which is weaker than the

saturated alkylene, cross-linked groups of the invention.

9. Turbak et al. discloses use of a solution of 3-15 weight percent lithium chloride in DMAC to polymerize cellulose. There is no teaching of the presence of a saturated hydrocarbon cross-linking agent.

DECLARANT declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code and that such willful, false statements may jeopardize the validity of the application or document or any registration resulting therefrom.

Signed at VENTURA, CALIFORNIA, this  
7<sup>th</sup> day of AUGUST 2003.

  
Wilson Hago

**Wilson Hago, Ph.D.**  
**52 North Ash Street**  
**Ventura, California 93001**  
**(805) 652-0368**

### ***Education***

- Ph.D., Physical Chemistry, Brown University, 1994.  
Thesis Title: Coadsorption of Alkalies and Hydrogen on W(100). Advisor: Peder J. Estrup.  
Research Proposal Doctoral Requirement: "A proposal to investigate the EL2 defect in GaAs (110) with the scanning tunneling microscope".
- Post-Baccalaureate, Organic Chemistry, City College of the City of New York, 1985-1986.
- A.B., Physics, Harvard University, 1985.

### ***Work Experience***

Senior Research Chemist, Zinc Matrix Power, 1997-present.

Expert on cellulose chemistry and on cellulosic membranes as separators. Invented recombinant separator for silver-zinc batteries. Research Manager. Invented the addition of additives to separator to control ion migration in silver-zinc batteries.

Postdoctoral Fellow, University of California at Santa Barbara, 1994-96.

Performed work in alkane activation using molecular beams. Studied the breakdown of hydrocarbons on Iridium surfaces.

### ***Publications***

1. Coadsorption of alkalies and hydrogen on W(100), Journal of Vacuum Science and Technology, A13(3), 1559 (1995).
2. Effect of preadsorbed oxygen on the direct dissociative chemisorption of ethane on Ir(110), Journal of Vacuum Science and Technology, A13(3), 1426 (1995).
3. The effect of preadsorbates on the dissociation dynamics of ethane on Ir(110)", Journal of Vacuum Science and Technology, A14(3), 1578 (1996).

### ***Skills***

Knowledgeable in theory and practice of :

Nuclear magnetic resonance (NMR),

High Pressure Liquid Chromatography (HPLC)

Fourier Transform Infrared Spectroscopy (FTIR)

Polymer Synthesis

Mass Spectrometry

Supersonic Molecular Beams

Auger Spectroscopy

Low Energy Electron Diffraction

## ***Patents Granted***

- 1 6,582,851 **T** Anode matrix
- 2 6,558,849 **T** Battery separator with copper-containing inorganic salt
- 3 6,541,160 **T** Battery separator with sulfide-containing inorganic salt

## ***Patent applications***

Pub. NO. Title

- 1      20030087157 Homogeneous separator
- 2      20020182512 Battery separator with fluoride-containing inorganic salt
- 3      20020182510 Recombinant separator
- 4      20020182489 Silver-zinc alkaline rechargeable battery (stacking order)